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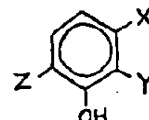
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(54) *m*-Alkyl phenol derivatives and  
their use in copper extraction

(57) Compounds of the formula



in which:

X represents a straight chain  
hydrocarbyl group containing from 9 to  
17 carbon atoms;

Y represents H or the group -CH=NOH,  
and

Z represents H or the group -CH=NOH  
or the group -CPh=NOH, provided that  
at least one of Y and Z represents H; or  
such compound containing at least one  
electronegative substituent in the  
aromatic ring,

may be prepared from anacardic acid  
and its analogues, and are useful for the  
extraction of copper from acidic  
aqueous solution.

GB 2 104 516 A

## SPECIFICATION

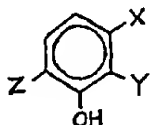
## Metal extraction

- 5 The compound (2-hydroxy 5-tertiary nonyl) acetophenone oxime which is commercially available in a hydrocarbon diluent, is useful for the extraction of copper from acidic media but is at present derived from petroleum the supply and price of which is liable to fluctuation.

Compounds have now been found which can extract relatively greater amounts of copper from acidic media and which can be produced from the replenishable source Cashew Nut-Shell Liquid (CNSL) and from related *Anacardium* species.

Accordingly the present invention comprises a compound of formula I.

20



wherein:-

- 25 X represents a straight chain hydrocarbyl group containing from 9 to 17 carbon atoms; when Z represents hydrogen, Y represents the group  $-\text{CH}=\text{NOH}$ ; and when Y represents hydrogen, Z represents the group  $-\text{CH}=\text{NOH}$  or  $-\text{CPh}=\text{NOH}$ .

Electronegative substituents, such as chloro, nitro and cyano groups, may also be present on the aromatic ring.

- 35 Compounds of formula I are preparable from the carbonyl precursors of the oximes by treatment with appropriate reagents e.g. hydroxylamine salts, typically the hydrochloride or sulphate in a solvent such as anhydrous pyridine, if necessary or preferred with the application of heat.

- 40 The present invention further includes within its scope carbonyl compounds IA which are compounds of formula I in which:-

X represents a straight chain hydrocarbyl group containing from 9 to 17 carbon atoms.

- 45 when Z represents hydrogen, Y represents  $-\text{CHO}$  and when Y represents hydrogen, Z represents  $-\text{CHO}$  or  $-\text{COPh}$ .

- Aldehyde precursors for compounds I in which Y represents  $-\text{CH}=\text{NOH}$  may be produced for example from 15:0, 15:1 and/or 15:2 anacardic acid IB, which are compounds of formula I in which  $\text{Z}=\text{H}$ ,  $\text{Y}=\text{CO}_2\text{H}$  and  $\text{X}=\text{C}_{15}\text{H}_{27}$ ,  $\text{C}_{15}\text{H}_{29}$  or  $\text{C}_{15}\text{H}_{31}$  by various methods. Such methods include

- 55 (1) Reduction e.g. hydride reduction to the corresponding alcohol followed by oxidation thereof to the aldehyde using for example periodate to produce a spirodienone which is subsequently ultraviolet irradiated or using pyridinium

- 60 chlorochromate or a pyridine/chromium trioxide mixture or  
(2) conversion of the acid to the corresponding acid chloride or anhydride or a mixture thereof by treatment with e.g. thionyl chloride followed by reduction of the product to the corresponding aldehyde by means for example of a suitable Rosenmund catalyst and hydrogen or by hydride.

Anacardic acid is available from natural CNSL as a mixture comprising the compounds IB and it may be desirable to partly or fully reduce the  $\text{C}_{15}$  side chain to produce the 15:1 or 15:0 compound or a mixture of such compounds prior to conversion to the 15:1 or 15:0 oximes which are preferred extractants. Full or partial reduction may be accomplished by hydrogenation using for example a catalyst of the Raney or Sabatier type or a palladium catalyst whereas partial reduction to the 15:1 acid can be effected by diimide in a solvent such as ethanol, the diimide being generated *in situ* suitably from hydrazine hydrate and air.

Aldehyde precursors for compounds I wherein Z represents the group  $-\text{CH}=\text{NOH}$  may be produced by oxidation of the corresponding alcohol using for example the reagents hereinbefore described, the alcohol being obtainable by reduction, typically hydride reduction, of the corresponding acid. Such an acid can be produced for example by a Kolbe reaction of cardanol, IC a compound of formula I in which  $\text{Z}=\text{H}$ ,  $\text{Y}=\text{H}$  and  $\text{X}=\text{C}_{15}\text{H}_{27}$ ,  $\text{C}_{15}\text{H}_{29}$  or  $\text{C}_{15}\text{H}_{31}$ , or an alkali metal salt thereof with carbon dioxide.

The present invention also includes within its scope compounds ID of formula I in which Z represents  $-\text{CO}_2\text{H}$ , Y represents hydrogen and X represents a straight chain hydrocarbyl group containing from 9 to 17 carbon atoms.

Alternatively, cardanol (IC) can be methylolated to produce a compound comprising a  $-\text{CH}_2\text{OH}$  group at the ring position para to the side chain by treatment with an alkylmagnesium halide e.g. ethyl magnesium bromide to give the phenoxymagnesium halide derivative of cardanol followed by treatment thereof with paraformaldehyde in an aprotic solvent such as hexamethyl phosphoric triamide. The methylolated product can then of course be readily oxidised to the corresponding aldehyde by methods such as those hereinbefore described.

In a further method for production of aldehyde precursors for oximes of formula I in which Z represents  $-\text{CH}=\text{NOH}$ , cardanol is converted to the phenoxymagnesium halide derivative which is treated with ethylorthoformate in an aprotic solvent such as tetrahydrofuran followed by hydrolysis of the intermediate acetal. In yet further methods for the production of the aldehyde cardanol, preferably 15:0 cardanol, is formylated by means of the Gatterman reaction, the product containing an impurity 3-pentadecyl-4-formyl phenol, or is subjected to the

The chemical formula appearing in the printed specification was submitted after the date of filing, the formula originally submitted being incapable of being satisfactorily reproduced.

This print takes account of replacement documents later filed to enable the application to comply with the formal requirements of the Patents Rules 1982.

Hoesch reaction.

The present invention also includes within its scope alcohols from which the hereinbefore described aldehydes may be obtained by oxidation thereof. Such alcohols, IE are compounds of formula I, in which: Z represents  $-\text{CH}_2\text{OH}$  when Y represents hydrogen Y represents  $-\text{CH}_2\text{OH}$  when Z represents hydrogen and X represents a straight chain hydrocarbyl group containing from 9 to 17 carbon atoms.

Electronegative ring substituents, such as chloro, nitro and cyano groups, enhance the chelation properties of the oxime product. Such substituents may be introduced, by conventional methods, onto the aromatic ring of either the acid or the alcohol. Such substituted acids and alcohols are within the scope of the invention.

Cardanol is obtainable from Technical Cashew Nut-Shell Liquid (CNSL) in which it is present in high concentration or Technical CNSL may be used *per se* generally after reduction for example as hereinbefore described for anacardic acid to give compounds containing 15:0 or 15:1 side chains or a mixture comprising such compounds.

Technical CNSL or cardanol may also be used as a starting material for the production of the ketone precursor or compounds I in which Z represents  $-\text{CPh}=\text{NOH}$ . After any desired reduction of the unsaturation in the side chain by methods such as those hereinbefore described, CNSL or Cardanol may be treated with a benzoyl halide, typically benzoyl chloride in the presence of a Lewis acid such as  $\text{AlCl}_3$  under conditions appropriate for Friedel-Crafts reaction to yield the precursor.

Different species of *Anacardium* yield anacardic acids containing hydrocarbyl side chains of different lengths. For example, an anacardic acid containing a  $\text{C}_{11}$  side chain may be obtained from the *Anacardium giganteum*, and particularly from the nut-shell of this species.

Extractants of formula 1 may be utilised for extraction of a metal such as copper by treatment of a solution or dispersion thereof in a water immiscible solvent (typically a non polar solvent such as kerosene), with a mildly acidic aqueous solution of the metal salt e.g.  $\text{CuSO}_4$  which generally contains salts of other metals such as nickel. Following agitation of the two phase system to promote extraction into the non-polar phase the phases are separated and the extracted metal salt is stripped therefrom, generally by treatment with an acid e.g. sulphuric acid at a concentration greater than that present in the aqueous phase before extraction e.g. 1.5 molar or stronger sulphuric acid. The present invention is illustrated by the following Examples:—

#### Example 1

(15:0)-Anacardic aldoxime (6-Pentadecylsalicylaldoxime)

(A) 15:0 Anacardic Alcohol

Anacardic acid consisting of a mixture of (15:0), (15:1), (15:2) and (15:3) - anacardic acids is extracted from natural cashew nut-shell liquid as described in Tyman and Lam, J. Chem. Soc., Perkin 1, 1981, 1924 and is obtained as an oil in 69% yield.

The oil (30g) in ethyl acetate (200  $\text{cm}^3$ ) containing

10% palladium-carbon catalyst (3.0g) is shaken with hydrogen at  $15 \text{ lb in}^{-2}$  at ambient temperature. After absorption of hydrogen (3,530  $\text{cm}^3$ ) the reduction is complete. The mixture is filtered to recover the catalyst and upon evaporation and recovery of the solvent, (15:0)-anacardic acid obtained in theoretical yield, m.p. 87-88°.

(15:0)-Anacardic acid (5.2g) in dry tetrahydrofuran (60  $\text{cm}^3$ ) is added dropwise over two hours to lithium aluminium hydride (2.5g) suspended in tetrahydrofuran (25  $\text{cm}^3$ ) and the stirred mixture is refluxed for two hours with monitoring by thin layer chromatography to indicate complete reduction. The excess hydride is decomposed by the addition of ethyl acetate and the acidified mixture is extracted with ether. Recovery gives a brown oil which upon crystallisation from light petroleum (40-60°) yields (15:0)-anacardic alcohol (6-pentadecylsalicyl alcohol) as cream coloured crystals, m.p. 60-62° (4.05g).

(B) 15:0 Anacardic Aldehyde

(15:0)-Anacardic alcohol (2.0g) in methanol (30  $\text{cm}^3$ ) is treated with a solution of sodium periodate (1.76g) in water (10  $\text{cm}^3$ ). After several minutes a thick yellow solid separates and the stirred mixture is warmed at 30°C for one hour to complete the reaction. Dilution with water and ethereal extraction gives a thick yellow oil which after crystallisation from light petroleum (40-60°) affords the spirodienone, 8 - pentadecyl - 1 - oxaspiro (2.5) octa - 5,7 - dien - 4 - one; m.p. 80-81°C (1.20g).

The spirodienone (1.08g) in ethanol is irradiated with ultraviolet light from a medium pressure Hanovia photochemical reactor during three hours. Concentration of the solution, ethereal extraction and crystallisation from light petroleum (40-60°) gives pale yellow of (15:0)-anacardic aldehyde (6-pentadecylsalicylaldehyde, h.p. 45-47° (1.01g).

(C) 15:0 Anacardic aldoxime

(15:0)-Anacardic aldehyde (0.41g) in pyridine (7  $\text{cm}^3$ ) is heated at 100°C with hydroxylamine hydrochloride (0.746g). Oximation is complete after five hours and the mixture is diluted with water, acidified with hydrochloric acid and ethereally extracted.

Concentration of the dried ethereal solution gives a greyish oil which after crystallisation from light petroleum (40-60°) furnished (15:0)-anacardic aldoxime as off-white prisms, m.p. 95-97°, (0.34g). Found: C, 75.45; H, 10.77; N, 3.80.  $\text{C}_{22}\text{H}_{37}\text{O}_2\text{N}$  requires C, 76.08; H, 10.66; N, 4.03%. It had the expected  $^1\text{H}$  NMR and IR absorption spectra.

#### Example 2

15:0 Anacardic aldoxime

The oxime is produced as described in Example 1 from 15:0 Anacardic aldehyde produced by a procedure in which oxidation of (15:0)-anacardic alcohol (2.0g) in dichloromethane (110  $\text{cm}^3$ ) with pyridinium chlorochromate (5.50g), prepared from chromium trioxide and 6M hydrochloric acid is followed by addition at 0° of pyridine according to Corey and Suggs (Tetrahedron Letters, 1975, 2647), during 90 minutes at ambient temperature. Filtration of the reaction mixture, concentration of the filtrate and crystallisation gives (15:0)-anacardic aldehyde identical with the product from the process described in

## Example 1.

## Example 3

## (A) 15:0 Anacardic aldehyde

- (15:0)-Anacardic acid (3.48g) in light petroleum, 40-60°C (20 cm<sup>3</sup>) containing pyridine (0.02g) is stirred and treated with thionyl chloride (0.62 cm<sup>3</sup>) at 30°. After two hours the mixture which becomes almost clear is filtered and the filtrate concentrated to give an oily product consisting of the acid chloride and the acid anhydride.

- The crude product from two such reactions (5g) in dry xylene (9.2 cm<sup>3</sup>) containing 5%-palladium barium sulphate catalyst (0.276g) and thiourea, catalyst poison, (0.055g) is heated at 140-150° and hydrogen passed into the mixture with the usual precautions. Hydrogen chloride soon appears in the exit gas and is collected in a water trap. When no more is produced the reaction is stopped, the reaction mixture is cooled, decolourising charcoal (1g) added and after filtration and concentration the residual material (4.60g) is crystallised from light petroleum (40-60°) to give (15:0)-anacardic aldehyde which is identical with the products from the reactions described in Examples 1 and 2.

## 25 (B) 15:0 Anacardic aldoxime

- The aldehyde is converted into the corresponding aldoxime as described in Example 1.

## Example 4

## (A) 15:0 Anacardic aldehyde

- A mixture of acid chloride and anhydride produced as described in Example 3 (1.0g), is reduced in diglyme (1.1 cm<sup>3</sup>) at -75°C with a t-butanol/lithium aluminium hydride complex, obtained by reacting dry t-butanol (0.6g) with lithium aluminium hydride (0.106g) in dry ether (5.6 cm<sup>3</sup>) and dissolving the precipitate in diglyme (2.2 cm<sup>3</sup>), to give (15:0)-anacardic aldehyde which is purified by crystallisation to give (0.69g).

- (B) 15:0 Anacardic aldoxime is produced from the aldehyde as described in Example 1.

## Example 5

## 4 - Pentadecylsalicylaldehyde ("15:0" - iso - anacardic aldoxime")

## (A) 4 - Pentadecylsalicylic acid

- The unsaturated product "iso-anacardic acid" 4-pentadecylsalicylic acid from the Kolbe reaction of the sodium salt of cardanol with carbon dioxide is hydrogenated to give the saturated compound in the following manner.

- The unsaturated acid, consisting of (15:0), (15:1), (15:2) and (15:3) constituents (20g) in ethanol (150 cm<sup>3</sup>) containing 10% palladium-carbon catalyst (2.0g) is hydrogenated until 2500 cm<sup>3</sup> of hydrogen has been absorbed. Filtration to recover the catalyst and concentration of the filtrate followed by crystallisation of the residual material gives 4-pentadecylsalicylic acid, (15:0)-iso-anacardic acid as greyish prisms, m.p. 96-97° (14.8g).

## (B) 4-Pentadecylsalicyl alcohol

- 4-Pentadecylsalicylic acid (5.2g) in tetrahydrofuran (60 cm<sup>3</sup>) is slowly added to stirred lithium aluminium hydride (2.5g) in dry tetrahydrofuran (25 cm<sup>3</sup>) and the mixture then stirred and refluxed for five hours to complete the reduction. Work-up by recovery of the tetrahydrofuran, addition of a little

ethyl acetate to the cooled residual material, followed by acidification and ethereal extraction yields the crude product which is crystallised from light petroleum (40-60°) to give 4-pentadecylsalicyl alcohol as off-white prisms, m.p. 66-68°, (4.05g).

## (C) 4-Pentadecylsalicylaldehyde

- 4-Pentadecylsalicyl alcohol (1.54g) in methanol (25 cm<sup>3</sup>) is treated with sodium periodate (1.30g) in water (8 cm<sup>3</sup>). A thick yellow solid soon separates and the mixture is warmed to 30° during five hours to complete the reaction which is indicated by monitoring with thin layer chromatography. Dilution of the mixture with water and ethereal extraction followed by concentration of the ethereal layer and crystallisation of the residue from light petroleum (40-60°) affords the "isomeric spiro epoxy dienone", m.p. 84-86°C, (0.82g).

- Irradiation of the spirodienone (0.72g) in ethanol with ultraviolet light in an Hanovia photochemical reactor during three hours and recovery and crystallisation of the product from light petroleum (40-60°) gives 4-pentadecylsalicylaldehyde as a pale yellow material, m.p. 50-54°, (0.61g).

## (D) 4-Pentadecylsalicylaldoxime

- Interaction of the aldehyde in pyridine solution with hydroxylamine hydrochloride at 100° during five hours yields 4-pentadecylsalicylaldoxime, "(15:0)-iso-anacardic aldoxime".

## Example 6

## 95 (A) 4-Pentadecylsalicylaldehyde

- Cardanol consisting of a mixture of (15:0)-cardanol, (15:1)-cardanol, (15:2)-cardanol and (15:3)-cardanol is obtained from the technical cashew nut-shell liquid by vacuum distillation in 70% yield.

- The mixed material in ethanol containing 10% palladium-carbon catalyst is hydrogenated until absorption of hydrogen ceased and the side-chain has been saturated as revealed by argentation thin layer chromatography and <sup>1</sup>H NMR absorption spectroscopy. The product is recovered by filtration, concentration of the filtrate and crystallisation of the residual material from light petroleum (40-60°) to give (15:0)-cardanol, containing a little (15:0)-cardol. (15:0)-Cardanol (10.2g) is added to an equivalent proportion of ethyl magnesium bromide in dry ethereal solution of which is observed when evolution of ethane ceases. Ethyl orthoformate (10 cm<sup>3</sup>) is added and the ether removed by distillation so that the temperature rises to 100°C. After refluxing for three hours, the mixture is decomposed with dilute hydrochloric acid and warmed to effect the hydrolysis of the intermediate acetal formed. Ethereal extraction and recovery gives a mixture of some unchanged (15:0)-cardanol and the formylated product, 4-pentadecylsalicylaldehyde which is separated by chromatography.

## (B) 4-Pentadecylsalicylaldoxime

- The aldehyde is converted into the corresponding aldoxime by the process described in Example 5.

## Example 7

## (A) 4-Pentadecylsalicylaldehyde

- Cardanol (2.0g) is converted as described in Example 6 to the phenoxy magnesium bromide derivative and refluxed in benzene (50 cm<sup>3</sup>) contain-

ing hexamethyl phosphoric triamide (1.2g) and para-  
formaldehyde (0.67g) for three hours. Acidification  
and ethereal extraction followed by drying and con-  
centration gives a pale brown oil consisting of some  
unchanged (15:0)-cardanol and  
4-pentadecylsalicylaldehyde which are separated by  
chromatography.

(B) 4-Pentadecylsalicylaldoxime

The aldehyde is converted into the corresponding  
aldoxime by the process described in Example 5.

Example 8

2-Hydroxy-4-pentadecyl benzophenone ketoxime

(A) 2-hydroxy-4-pentadecylbenzophenone

(15:0)-Cardanol (2.0g) in dry pyridine (5 cm<sup>3</sup>) is  
treated with benzoyl chloride (1.5 cm<sup>3</sup>) and the mix-  
ture warmed for five hours at 100°C. The cooled mix-  
ture is diluted with water, basified with dilute  
sodium hydroxide solution, and the precipitated  
(15:0)-cardanol benzoate collected by filtration,  
washed with water until neutral and dried to give  
2.785g.

3-Pentadecylphenyl benzoate (1.366g) is finely  
powdered, mixed with pulverised anhydrous  
aluminium chloride (0.616g) and the intimate mix-  
ture is heated to 165° during fifteen minutes. After  
hydrogen chloride evolution has ceased, the cooled  
mixture is treated with water, concentrated hydroch-  
loric acid added, and the whole thoroughly stirred to  
decompose the aluminium complex. The product is  
ethereally extracted and the dried ethereal layer is  
concentrated to give the crude product (1.348g)  
which possesses a greenish ferric chloride reaction.  
Chromatographic purification and crystallisation  
from light petroleum (40-60°) affords 2-hydroxy-4-  
pentadecylbenzophenone, m.p. 46-47°.

75	Moles of reagent oxime used
	Equivalent to moles copper
	Equivalent to g copper
	Max. theoretical loading
	Copper extracted (g)
	Copper initially present (g)
80	% copper extracted
	% theoretical loading of copper
	% recovery of copper

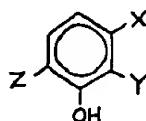
Both reagents are stable to the acidic conditions of  
recovery as assessed by thin layer chromatography.

Example 10

In an extraction process similar to that of Example  
9 in which nickel chloride is present with copper sul-  
phate in equimolar strength, analysis by atomic  
absorption indicates that anacardic aldoxime (like  
the ketoxime reagent) extracts copper specifically.

CLAIMS

1. A compound useful for extraction of copper  
from acidic media of the formula



(B) 2-hydroxy-4-pentadecyl benzophenone  
ketoxime

Conversion to 2-hydro-4-pentadecylben-  
zophenone oxime is effected in pyridine solution  
with hydroxylamine hydrochloride as described in  
Example 1 or 5 to give the product, m.p. 51-3°C.

Example 9

The extraction properties of (15:0)-anacardic  
aldoxime for copper ions are compared with those  
of the commercial extractant, the oxime of 2-acetyl-  
4-nonyl phenol (SME 529). The following solutions  
are used.

- A Standard copper sulphate solution, 5.715g/litre  
of CuSO<sub>4</sub> · 5H<sub>2</sub>O
- B Anacardic aldoxime, 1.25% w/w, 3.6 x 10<sup>-3</sup>  
moles/100 cm<sup>3</sup> of petroleum (100-120)
- C 2-Acetyl-4-nonyl phenol ketoxime, 1.25% w/w,  
4.5 x 10<sup>-3</sup> moles/100 cm<sup>3</sup> of petroleum  
(100-120°)
- D 1.50 M aqueous sulphuric acid.

Solution B (20 cm<sup>3</sup>) is thoroughly mixed with Solu-  
tion A (10 cm<sup>3</sup>) and similarly Solution C (20 cm<sup>3</sup>) is  
mixed with Solution A (10 cm<sup>3</sup>). Excess of copper ion  
is present to ensure, if possible, maximum loading  
of oxime compounds. The pH was 5. Both solutions  
are thoroughly shaken and after emulsion separa-  
tion has occurred, the lower copper sulphate aque-  
ous layer is removed and analysed for copper sul-  
phate by iodometric titration. The following results  
at the extraction stage are obtained on the basis that  
1g atom of copper is associated with 2 moles of the  
oxime derivative. Each of the organic solutions are  
finally washed with Solution D (2 x 10 cm<sup>3</sup>) to  
recover the copper extracted.

Anacardic Aldoxime	
7.20 x 10 <sup>-4</sup>	9.00 x 10 <sup>-4</sup>
3.60 x 10 <sup>-4</sup>	4.50 x 10 <sup>-4</sup>
2.29 x 10 <sup>-2</sup>	2.86 x 10 <sup>-2</sup>
2.29 x 10 <sup>-2</sup>	2.86 x 10 <sup>-2</sup>
2.33 x 10 <sup>-2</sup>	1.27 x 10 <sup>-2</sup>
5.715 x 10 <sup>-2</sup>	5.715 x 10 <sup>-2</sup>
40.7	22.2
100	44.4
91	100

in which:

X represents a straight chain hydrocarbyl group con-  
taining from 9 to 17 carbon atoms;

Y represents H or the group -CH=NOH, and

Z represents H or the group -CH=NOH or the group  
-CPh=NOH, provided that at least one of Y and Z  
represents H; or such compound containing at least  
one electronegative substituent in the aromatic ring.

2. A method of extracting copper from an acidic  
aqueous solution containing copper comprising:  
treating such a solution with a solution of a com-  
pound according to claim 1 in a water immiscible  
solvent;

separating the aqueous phase from the water  
immiscible phase; and

extracting the copper from the water immiscible  
phase.

3. A compound substantially as hereinbefore described with reference to any of the Examples.

4. A method according to claim 2 substantially as hereinbefore described with reference to Example 9 or 10.

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